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### P. . ENT COOPERATION TREA From the INTERNATIONAL BUREAU PCT NOTIFICATION OF THE RECORDING KAZI, Ilya Mathys & Squire OF A CHANGE 100 Grays Inn Road (PCT Rule 92bis.1 and London WC1X 8AL Administrative Instructions, Section 422) ROYAUME-UNI Date of mailing (day/month/year) 22 March 2001 (22.03.01) IMPORTANT NOTIFICATION Applicant's or agent's file reference International filing date (day/month/year) IK/20844 26 August 1999 (26.08.99) International application No. PCT/GB99/02822 1. The following indications appeared on record concerning: the common representative the agent State of Residence the inventor State of Nationality the applicant Χl GB GB Name and Address JUMIK TECHNOLOGIES LIMITED Telephone No. Office 791 Old Brompton Street Facsimile No. London SW7 3DQ United Kingdom Teleprinter No. 2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning: the residence State of Residence the name X the person State of Nationality GB Name and Address PVAXX TECHNOLOGIES LIMITED Telephone No. Kemble Business Park North Malmesbury SN16 9SH Facsimile No. United Kingdom Teleprinter No. Further observations, in necessary. A power of attorney signed on behalf of the person appearing in Box 2 above is required. 3. Further observations, if necessary: 4. A copy of this notification has been sent to: the designated Offices concerned the elected Offices concerned the receiving Office the International Searching Authority other: the International Preliminary Examining Authority Authorized officer The International Bureau of WIPO R. Chrem 34, chemin des Colombettes Telephone No.: (41-22) 338.83.38 1211 Geneva 20, Switzerland

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### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

		The file reference	T		
Applicants IK/20844	_	ent's file reference	FOR FURTHER ACT	See N Prelin	lotification of Transmittal of International ninary Examination Report (Form PCT/IPEA/416)
Internation		ication No.	International filing date (day	//month/year)	Priority date (day/month/year)
PCT/GB			26/08/1999	,	26/08/1998
International CO8K13/		nt Classification (IPC) or na	ational classification and IPC		
Applicant					
JUMIK T	ECH	NOLOGIES LIMITED	et al.		•
and is	trans	smitted to the applicant a	according to Article 36.	-	International Preliminary Examining Authority
2. This	REPO	PRT consists of a total of	4 sheets, including this c	over sheet.	
b (:	een a see R	mended and are the ba	sis for this report and/or sh 07 of the Administrative In	neets containi	ription, claims and/or drawings which have ng rectifications made before this Authority der the PCT).
1	×	Basis of the report	ating to the following items	:	
		•	minion with report to pave	ultur invantiva	oton and industrial applicability
III IV		Lack of unity of inventi-	· -	ally, inventive	step and industrial applicability
v	Ø	Reasoned statement u		ard to novelty	, inventive step or industrial applicability;
VI		Certain documents cit	ed		
VII	$\boxtimes$	Certain defects in the i	nternational application		
VIII	Ø	Certain observations o	n the international applica	tion	
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International application No. PCT/GB99/02822

### I. Basis of the report

1.	resp the	onse to an invitation	rawn on the basis of (substitu on under Article 14 are referre o not contain amendments (Fi	ed to in this repo	rt as "originally filed	ed to the receiving Office in d" and are not annexed to
	1-14	1	as originally filed			
	Clai	ms, No.:				
	1-30	)	as received on	25/09/2000	with letter of	25/09/2000
2.	lang	juage in which the i	uage, all the elements markenternational application was available or furnished to this A	filed, unless othe	erwise indicated un	nder this item.
		the language of a the language of pu	translation furnished for the publication of the international attranslation furnished for the p	ourposes of the i	nternational search er Rule 48.3(b)).	n (under Rule 23.1(b)).
3.			leotide and/or amino acid s y examination was carried ou			
		contained in the in	ternational application in writ	ten form.		
		filed together with	the international application i	n computer read	lable form.	
		furnished subsequ	ently to this Authority in writte	en form.		
		furnished subsequ	ently to this Authority in com	puter readable f	orm.	
			t the subsequently furnished pplication as filed has been for		e listing does not g	go beyond the disclosure in
		The statement tha listing has been fu	t the information recorded in rnished.	computer reada	ble form is identica	l to the written sequence
4.	The	amendments have	resulted in the cancellation	of:		
		the description,	pages:			
		the claims,	Nos.:			
		the drawings,	sheets:			
5.			en established as if (some of beyond the disclosure as filed		nts had not been m	ade, since they have been



(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

- 6. Additional observations, if necessary:
- V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- 1. Statement

Novelty (N)

Yes:

Claims 1-30

No:

Claims

Claims

Inventive step (IS)

Yes: No:

Claims 1-30

Industrial applicability (IA)

Yes:

Claims 1-30

No: Claims

2. Citations and explanations see separate sheet

### VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted: see separate sheet

### VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made: see separate sheet



### **EXAMINATION REPORT - SEPARATE SHEET**

- The following documents (D) are referred to in this report: ı
  - D1 IE-A 97 280
  - D2 US-A 4 206 101
  - D3 WO 97/09379
- The new set of claims is fully supported by the application as filed and meets the 11 requirements of Article 34 (2)(b) PCT
- The documents cited in the search report, in particular D1-D3, disclose extrudable Ш PVA compositions which contain a fatty acid amide and optionally a plasticizer and/or a filler. These compositions are however converted to tablets or granulate suitable as feedstock for use in extrusion by a method itself involving extrusion. There is no disclosure of cold-pressing the tablets or pellets. The applicant has made plausible that the feedstock of the present independent claims 1 and 16, the method of the present independent claims 23 and 29 and the use according to claim 30 present specific advantages and solve the problem of extruding PVA based compositions while avoiding degradation of the polymer.
- The documents cited in the search report do not disclose or suggest the IV combination of PVA and a superadsorbent filler. Claim 27 does not require the incorporation of a lubricant or moisture and does not require that the feedstock be coldpressed and is thus much broader than the other claims. In the absence of experimental evidence, it not plausible that the compositions of claim 27, without further additives, can be extruded without significant polymer degradation of the PVA. This claim thus lacks support in the description and does not meet the requirements of Article 6 PCT.

It cannot be excluded that in some Designated States an objection for lack of unity will be raised might, since claim 27 is directed to a problem which is not related to the problem underlying claims 1-26, 29 and 30 and shares no technical features with these claims except for the -conventional- use of PVA as a matrix.

The description is not adapted to the present set of claims.

### **CLAIMS**

- 1. A polymer feedstock in the form of a cold-pressed tablet or pellet for use in extrusion of an extruded product containing PVA, the cold-pressed tablet or pellet comprising a blend of PVA and up to 5% lubricant by weight to render the blend extrudable.
- 2. A polymer feedstock according to Claim 1 wherein the lubricant comprises a fatty acid amide.

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- 3. A polymer feedstock according to Claim 2 wherein the fatty acid amide is a straight or branched  $C_{12}$ - $C_{24}$  fatty acid amide.
- 4. A polymer feedstock according to Claim 2 or 3 further comprising a plasticiser.

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- 5. A polymer feedstock according to Claim 4 wherein the plasticiser is selected from ethylene glycol, glycerine, glycerol, triethylene glycol, low molecular weight polyethylene glycols and  $C_2$ - $C_8$  amides.
- 20 6. A polymer feedstock according to any of Claims 1 to 5 comprising up to 20% plasticiser, by weight.
  - 7. A polymer feedstock according to any preceding claim further comprising a filler.
  - 25 8. A polymer feedstock according to Claim 7 wherein the filler comprises inert, inorganic material.
    - 9. A polymer feedstock according to Claim 7 wherein the filler comprises a superabsorbent material.

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10. A polymer feedstock according to Claim 8 or 9 wherein the filler comprises both

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an inert, inorganic material and superabsorbent material.

- 11. A polymer feedstock according to Claim 8, 9 or 10 wherein the inorganic filler comprises calcium carbonate.
- 12. A polymer feedstock according to any preceding claim comprising, by weight, up to 50% filler, up to 5% lubricant and up to 20% of a plasticizer.
- 13. A polymer feedstock according to Claim 12 comprising, by weight, 5-50% filler, 0.5 to 2.5% lubricant, 5 to 15% plasticizer and 40-80% PVA.
  - 14. A polymer feedstock according to any of Claim 1-13, further comprising an additional lubricant component to reduce adhesion of the polymer feedstock to surfaces.
- 15 15. A polymer feedstock according to Claim 14 wherein the additional lubricant comprises a stearate.
  - 16. A PVA-containing polymer feedstock in the form of a cold-pressed tablet or pellet comprising, by weight:-
- 20 40 to 80% PVA;

5 to 50% filler;

5 to 15% plasticizer; and

0.5 to 2.5% lubricant.

25 17. A polymer feedstock according to Claim 16, comprising, by weight:-

40 to 70% PVA;

20 to 50% filler;

8 to 15% plasticizer;

0.5 to 1.5% internal lubricant; and

30 0.0001 to 0.1% external lubricant.

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- 18. A polymer feedstock according to Claim 16 or 17, wherein a fatty acid amide is provided as internal lubricant.
- 19. A polymer feedstock according to Claim 16, 17 or 18, wherein stearate is provided as external lubricant.
  - 20. A polymer feedstock according to Claim 17 comprising, by weight:-

50 to 60% PVA;

30 to 40% stearate-coated calcium carbonate;

10 8 to 15% glycerol;

0.5 to 1.5% octadecanamide; and

0.0001 to 0.1% zinc stearate.

- 21. A polymer feedstock according to any preceding claim prepared substantially without melting of the PVA.
  - 22. A polymer feedstock according to any preceding claim having a moisture content less than about 10% by weight but greater than 0.01% to bind the pellets or tablets.
- 23. A method of making a PVA-containing polymer feedstock comprising blending PVA with a lubricant, said lubricant including a fatty acid amide in the presence of an amount of moisture sufficient to bind the polymer feedstock into tablets or pellets upon cold pressing and cold pressing the feedstock into tablets or pellets.
- 25 24. A method according to Claim 23 comprising blending, in a high speed blender, PVA with up to 5% by weight of lubricant.
  - 25. A method according to any of Claims 22 to 24 comprising adding moisture to the components to be blended.
  - 26. A method according to any of Claims 23 to 25 wherein PVA and lubricant are fed

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into a high speed mixer gravimetrically.

- 27. A method of making a PVA-containing polymer feedstock comprising blending PVA and a filler, wherein the filler comprises a superabsorbent material.
- 28. A method according to Claim 27 comprising blending PVA and a superabsorbent material in the presence of sufficient moisture to bind the polymer feedstock into tablets or pellets upon cold pressing.
- 10 29. A method of extruding a PVA-containing polymer feedstock comprising blending PVA and at least one of a lubricant and a filler to form a feedstock for an extrusion process, the feedstock being in the form of pellets or tablets, and extruding the feedstock into a product, wherein the feedstock is prepared substantially without melting of the PVA.
- 30. Use of a fatty acid amide in an amount of 0.5% to 1.5% by weight as an internal lubricant together with use of an external lubricant in an amount of 0.0001 to 0.1% by weight in the manufacture of a cold-pressed polymer feedstock containing PVA and up to 50% filler wherein the feedstock is manufactured by cold pressing into pellets or tablets using residual moisture to bind the pellets or tables and substantially without melting of the PVA.





(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	f Transmittal of International Search Report				
IK/20844	ACTION (Form PC1/ISA/2	20) as well as, where applicable, item 5 below.			
International application No. International filing date (day/month/year) (Earliest) Priority Date (day/month/year)					
PCT/GB 99/02822	26/08/1999	26/08/1998			
Applicant					
NIMIN TERMINOLOGISC LIMITE	6 . 1				
JUMIK TECHNOLOGIES LIMITE	vet al.				
This International Search Report has been according to Article 18. A copy is being tra	n prepared by this International Searching Auth ansmitted to the International Bureau.	nority and is transmitted to the applicant			
This International Search Report consists	of a total of sheets.				
It is also accompanied by	a copy of each prior art document cited in this	report.			
Basis of the report					
a. With regard to the language, the	international search was carried out on the bas ess otherwise indicated under this item.	sis of the international application in the			
the international search w Authority (Rule 23.1(b)).	ras carried out on the basis of a translation of the	ne international application furnished to this			
		ternational application, the international search			
was carried out on the basis of the contained in the internation	onal application in written form.				
filed together with the inte	rnational application in computer readable forn	n.			
furnished subsequently to	this Authority in written form.				
furnished subsequently to	this Authority in computer readble form.				
	osequently furnished written sequence listing do is filed has been furnished.	oes not go beyond the disclosure in the			
the statement that the info	ormation recorded in computer readable form is	s identical to the written sequence listing has been			
2. Certain claims were fou	nd unsearchable (See Box I).				
3. Unity of invention is lac	king (see Box II).				
4. With regard to the title,					
4. With regard to the title,  X the text is approved as su	ibmitted by the applicant.				
	thed by this Authority to read as follows:				
	·				
5. With regard to the abstract,					
the text is approved as su	ibmitted by the applicant.				
the text has been establis within one month from the	shed, according to Rule 38.2(b), by this Authority address of mailing of this international search rep	ty as it appears in Box III. The applicant may, oort, submit comments to this Authority.			
6. The figure of the drawings to be publ	lished with the abstract is Figure No.				
as suggested by the appli	icant.	None of the figures.			
because the applicant fail	ed to suggest a figure.				
because this figure better	characterizes the invention.				

International Application No.

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08K13/02 C08L29/04

//(C08K13/02,5:20,3:26)

According to International Patent Classification (IPC) or to both national classification and IPC

### B. FIELDS SEARCHED

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
US 4 206 101 A (WYSONG ROBERT D) 3 June 1980 (1980-06-03) column 4, line 50 -column 5, line 1; claims 1,12; example 1	1-5,33
IE 970 280 A (SOLPLAX LIMITED) 24 September 1997 (1997-09-24) examples	1,2,5,8, 24,25
WO 97 09379 A (SOLPLAX LTD.) 13 March 1997 (1997-03-13) claims	1,2,5,8, 24,25
WO 96 20973 A (ENVIRONMENTAL PACKING L P) 11 July 1996 (1996-07-11) claims 1,7,8; examples	1,8-12
-/	
	3 June 1980 (1980-06-03) column 4, line 50 -column 5, line 1; claims 1,12; example 1  IE 970 280 A (SOLPLAX LIMITED) 24 September 1997 (1997-09-24) examples  WO 97 09379 A (SOLPLAX LTD.) 13 March 1997 (1997-03-13) claims  WO 96 20973 A (ENVIRONMENTAL PACKING L P) 11 July 1996 (1996-07-11) claims 1,7,8; examples

X Further documents are listed in the continuation of box C.	χ Patent family members are listed in annex.
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Date of the actual completion of the international search  14 December 1999	Date of mailing of the International search report $22/12/1999$
Name and mailing address of the ISA  European Patent Office, P.B. 5818 Patentlaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax: (+31-70) 340-3016	Authorized officer  Engel, S

International Application No
T/GB 99/02822

	tion) DOCUMENTS CONSIDERED TO BE RELEVANT	To-to-to-to-to-to-to-to-to-to-to-to-to-to
ategory °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to daim No.
(	US 3 713 965 A (WIDIGER A ET AL) 30 January 1973 (1973-01-30)	1-3,9, 10, 13-15, 18,19
	column 1, line 68 -column 2, line 46; claims 1,4	
x	US 3 470 122 A (RIDGEWAY LEE R ET AL) 30 September 1969 (1969-09-30) column 3, line 4 - line 13; claims 1-3	1-3,9,10

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nation on patent family members

International Application No
T/GB 99/02822

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				GB	1113082 A	

### **PCT**





INTERNATIONAL APPLICATION PUBLIS	HED U	JNDER THE PATENT COOPERATION	ON TREATY (PCT)
(51) International Patent Classification 7:		(11) International Publication Number:	WO 00/12615
C08K 13/02, C08L 29/04 // (C08K 13/02, 5:20, 3:26)	A1	(43) International Publication Date:	9 March 2000 (09.03.00)
(21) International Application Number: PCT/GB  (22) International Filing Date: 26 August 1999 (  (30) Priority Data: 26 August 1998 (26.08.98)  (71) Applicant (for all designated States except US): JUMI. NOLOGIES LIMITED [GB/GB]; Office 791, Old I. Street, London SW7 3DQ (GB).	26.08.9 C K TEC	BR, BY, CA, CH, CN, CR, C ES, FI, GB, GD, GE, GH, GM, KE, KG, KP, KR, KZ, LC, LK, MG, MK, MN, MW, MX, NO, SE, SG, SI, SK, SL, TJ, TM, T VN, YU, ZA, ZW, ARIPO pater SD, SL, SZ, UG, ZW), Eurasian KZ, MD, RU, TJ, TM), Europea DE, DK, ES, FI, FR, GB, GR,	U. CZ, DE. DK, DM, EE, HR, HU, ID, IL, IN, IS, JP, LR, LS, LT, LU, LV, MD, NZ, PL, PT, RO, RU, SD, FR, TT, UA, UG, US, UZ, at (GH, GM, KE, LS, MW, patent (AM, AZ, BY, KG, in patent (AT, BE, CH, CY, IE, IT, LU, MC, NL, PT,
<ul> <li>(72) Inventor; and</li> <li>(75) Inventor/Applicant (for US only): STEVENS, He [GB/GB]; Ballacriy Manor, Colby, Isle of Man (C</li> <li>(74) Agents: KAZI, Ilya et al.; Mathys &amp; Squire, 100 (Road, London WC1X 8AL (GB).</li> </ul>	GB).	With international search report	

(54) Title: PVA-CONTAINING COMPOSITIONS

### (57) Abstract

An extrudable PVA-containing composition is disclosed, which enables PVA articles to be manufactured on conventional extrusion apparatus. The composition includes a lubricant, such as a fatty acid amide, most preferably octodecanamide and is most preferably formed into pellets by cold pressing. The compositions are suitable for manufacturing a variety of products.

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- 1 -

### **PVA-CONTAINING COMPOSITIONS**

The present invention relates to PVA-containing compositions, particularly filled compositions, to methods of making such compositions and to methods of extruding PVA-containing compositions.

There is currently a significant and increasing demand for bio-degradable polymers, to replace non bio-degradable polymers which take up an ever greater amount of disposal space, such as land-fill, or need to be incinerated.

Polyvinyl alcohol (PVA) is one bio-degradable polymer which is in widespread use. As a film, PVA shows a high degree of impermeability to a number of gases. Incorporated into adhesive, PVA shows high adhesive strength. PVA has significant potential in a wide range of products. However, the applications of PVA have been limited because there is currently no formulation of PVA or PVA-containing composition which can readily be extruded.

While attempts have been made to extrude articles from PVA, it has been found that melt extruded PVA becomes very unstable and significant residues become adhered to the surfaces of the extrusion apparatus, requiring very careful control of process conditions, specialised extrusion apparatus and frequent shut down of the extrusion process and purging of the apparatus.

It is known to polymerize ethylene with vinyl alcohol to provide a copolymer of ethylenevinyl alcohol, in an attempt to retain the desirable properties of PVA whilst simultaneously obtaining an extrudable polymer. One such copolymer is marketed under the trade mark SOARNOL (registered trade mark of the Nippon Synthetic Chemical Industry Co, Limited). However, it is found that this copolymer has effectively lost all of the advantageous properties of PVA and is not an effective alternative to an extrudable PVA.

A chemically modified PVA is also known and sold as VINEX (registered trade mark of Air

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Products). This suffers, however, from the fact that some of the advantageous PVA properties are adversely affected by the chemical modification.

The search for bio-degradable polymers is acute in relation to certain sanitary products such as diapers and sanitary towels. However, these products typically comprise superabsorbent materials and at present such materials are only available in combination with conventional non bio-degradable polymers.

The present invention is concerned, at least in preferred embodiments, with providing PVA10 containing compositions, methods of manufacturing the same and methods of extruding PVA-containing compositions which overcome or at least ameliorate some of the disadvantages identified in the art.

Accordingly, a first aspect of the invention provides a PVA-containing composition comprising a blend of PVA and an amount of lubricant effective to render the blend extrudable.

The lubricant preferably comprises a fatty acid amide and the composition may advantageously further comprise a plasticiser, the resultant blend being suitable for extrusion in conventional melt-extrusion apparatus. In embodiments of the invention, a PVA-containing composition comprises up to 20%, especially 5 to 15%, plasticizer and up to 5%, especially 0.5 to 2.5%, lubricant, by weight.

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The composition of the present invention is thus an extrudable PVA-containing polymer, typically with a flexural modulus similar to other extrudable polymers. Known advantageous properties of PVA, such as its high tensile strength, good puncture resistance, and good barrier characteristics are retained in an extrudable composition which can be processed on current extrusion lines, blow-moulders and injection moulders without modification, and without the processing problems such as thermal degradation and high temperature cross linking, observed in attempts to extrude articles using hitherto known PVA-containing compositions.

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In embodiments of the invention, extrudable PVA-containing compositions have been obtained comprising fully hydrolysed PVA as well as partially hydrolysed PVA, and including PVA that is 70% or more hydrolysed.

- Further embodiments of the invention provide PVA-containing compositions in which the molecular weight of the PVA varies from around 20,000, in some cases from around 10,000, to greater than 150,000. Generally, the application of the present invention is not limited to PVA of any particular percent hydrolysis nor of any particular molecular weight.
- A second aspect of the invention provides a filled PVA-containing composition comprising a blend of PVA plus filler. In this composition, the filler is effectively a bulking agent and is relatively inexpensive compared to the PVA, thus a composition is obtained retaining advantageous properties of PVA whilst being cheaper per unit weight to produce.
- In both aspects, the invention preferably provides a PVA-containing composition in the form of pellets or tablets. These preferably have a size of between about 1mm and 5mm in diameter and are suitably obtained by a method of the invention in which PVA, preferably substantially uncompounded and in powder or granular form, is mixed with at least one of lubricant and filler, and optionally other compounding ingredients, and cold pressed to form a tablet or pellet. This cold-pressing method is substantially as described in WO-A-98/26911 in relation to different polymers.

With this method, the ingredients can be uniformly mixed and compounded in the correct proportions for the final product, and made into a form that is easier to handle than powder. In addition, since the mixture is cold pressed rather than melted, problems of thermal degradation or variation in properties are reduced.

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References to "cold pressing" are intended to imply applying pressure to the powder to cause agglomeration without substantial melting of the polymer. The temperature is preferably less than about 100 degrees Celsius, more preferably less than about 70-80 degrees Celsius. Preferably heat is not applied directly to the mixture. However, heat may be generated by

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mixing and/or pressing, and this may be allowed to warm the mixture, or cooling may be provided. It is permissible for some heat to be applied directly if necessary to facilitate binding. Some melting of the polymer or other ingredients may occur, particularly on a microscopic scale as a result of pressure at the interface between adjacent particles, but it is important that the bulk of the polymer granules do not liquefy.

Preferably, the mixture is compressed in discrete quantities into tablets or pellets, for example in a tablet press; this may provide more reliable agglomeration.

- 10 However, it is possible for the mixture to be extruded under pressure through an orifice, the pellets forming following exit of the mixture from the orifice. In such a case, binder or moisture may be added to the mixture, preferably as the mixture is extruded, or after preliminary mixing of the constituents.
- 15 Preferably, the mixture is substantially dry, but includes sufficient residual moisture to bind the tablets or pellets on pressing. This avoids the need to add a separate binder, and allows the tablets or pellets to be broken more easily as they enter the extruder simply by drying them further. When water is relied upon for binding, it is desirable to ensure that the tablets or pellets are dried prior to extruding, for example at between 40 and 80 degrees Celsius for 4-8 hours; this alleviates problems of vapour formation during extrusion. The moisture content is desirable to form the tablets, but after forming, the tablets should in most cases retain their shape even when dried.
  - Preferably, the moisture content is less than about 10% by weight, and preferably greater than 0.01% by weight, more preferably less than about 5%. If the pellets or tablets are extruded or otherwise formed at relatively low pressures, however, the moisture content may be about 20% or even higher to ensure binding. If the pellets are too moist, they may tend to stick together, inhibiting flow, and drying after forming may be desirable.
- 30 The moisture may comprise residual moisture in the polymer or in one or more of the compounding ingredients. In this way, moisture can be provided automatically simply by

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not drying the ingredients fully prior to mixing.

It is preferred that the filled PVA-containing composition is extrudable, and hence preferred that the filled PVA-containing composition further comprises an effective amount of a lubricant to render the composition extrudable. The lubricant suitably comprises a fatty acid amide or a plasticiser or a mixture of both a fatty acid amide and a plasticiser.

The lubricant in compositions of the invention is provided so as to improve the thermal stability of the composition under extrusion. The lubricant may be referred to as an internal lubricant, in that its function is to improve lubrication between the polymer chains. Compositions of the invention may be intended for use in or in connection with food stuffs, and hence it is preferred that the lubricant is approved for food use. It is further preferred that the lubricant is a fatty acid amide, and particularly a straight or branched  $C_{12}$ - $C_{24}$  fatty acid amide, more particularly  $C_{16}$ - $C_{20}$ . Particularly and surprisingly good results are obtained if the lubricant comprises stearamide, a straight chain  $C_{18}$  fatty acid amide.

The compositions of the invention typically further include a plasticiser, to lower the melt temperature of the polymer under extrusion. The plasticiser may suitably be selected from glycerine, ethylene glycol, triethylene glycol, low molecular weight polyethylene glycols and low molecular weight amides. A particularly preferred plasticiser comprises or consists of glycerol.

While the proportion of components present may vary in compositions of the invention, embodiments of the invention generally comprise, by weight, up to 50% filler and up to 5% lubricant. More particular embodiments of the invention comprise, by weight, 5-50% filler, 40-80% PVA, up to 5% lubricant and 5-20% plasticizer.

Compositions of the invention are thus extrudable, but it may be that ease of extrusion depends partly upon the state of the extrusion apparatus, in particular the roughness of surfaces in the apparatus. In further embodiments of the invention, the compositions comprises an additional lubricant, which may be referred to as an external lubricant, to

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prevent or reduce adhesion of the composition to such surfaces. This external lubricant is typically present at 0.001 to 0.1% by weight of the composition and preferably approved for food use, in the same way as the internal lubricant. Stearate salts are suitable external lubricants, in particular magnesium, lead or zinc stearates.

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Fillers for incorporation into compositions of the invention may suitably be selected from conventional polymer fillers. Typically the filler is an inert, inorganic material and a particularly preferred filler is or comprises talc, calcium carbonate. In a specific embodiment of the invention, this talc may be micronised, such as in particles with a mean size of about 20 microns and may further be coated, such as with a stearate. One such coated, micronised talc is available from Croxton and Gary as 90T (trade mark of Croxton and Gary).

It is further an option for the filler to comprise or consist of a superabsorbent material. In a specific embodiment of the invention, described in an example below, a filled PVA-containing composition comprises a superabsorbent which contains cross-linked sodium polyacrylate (available from Alloid Colloids as SALISORB CL31 (registered trade mark)). Filled compositions of the invention may nevertheless contain superabsorbent material of substantially any type. Incorporation of such superabsorbent material confers the particular advantage that sanitary products such as diapers and sanitary towels, hitherto major components of waste storage sites such as land-fill sites, may now be made of or comprise a significant proportion of bio-degradable material.

A number of water-absorbent compositions are known and suitable for use as the filler in embodiments of the invention. For example, US Patent numbers 3,954,721 and 3,983,095 disclose preparations for derivatives of copolymers of maleic anhydride with at least one vinyl monomer in fibrous form. The fibrous copolymers are rendered hydrophilic and water-swellable by reaction with ammonia or an alkali metal hydroxide. US Patent No. 3,810,468 discloses lightly cross-linked olefin-maleic anhydride copolymers prepared as substantially linear copolymers and then reacted with a diol or a diamine to introduce cross-linking. The resultant lightly cross-linked copolymers are treated with ammonia or

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an aqueous or alcohol solution of an alkali metal hydroxide. US Patent No. 3,980,663 describes water-swellable absorbent articles made from carboxylic polyelectrolytes via cross-linking with glycerine diglycidyl ether. These patents are incorporated herein by reference.

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European Published Application No. 0 268 498 (incorporated herein by reference) describes a water-absorbent composition formed by causing a substantially linear polymer of water-soluble ethylenically unsaturated monomer blends comprising carboxylic and hydroxylic monomers to cross-link internally.

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Further examples of water-absorbent compositions are those produced from a copolymer of an  $\alpha$ , $\beta$  unsaturated monomer having at least one pendant unit selected from a carboxylic acid group and derivatives thereof and a copolymerisable monomer. A proportion of the pendant units are present in the final copolymer as the free acid and a proportion as the salt of the acid. These copolymers are capable of being cross-linked, either internally or with a variety of cross-linking agents, to form the water-swellable composition. Examples of water-swellable compositions of this type can be found in US Patent Nos 4,616,063, 4,705,773, 4,731,067, 4,743,244, 4,788,237, 4,813,945, 4,880,868 and 4,892533 and European Patent Nos 0 272 074 and 0 264 208 and European Published Application No. 0 436 514 which are incorporated herein by reference.

Derivatives of carboxylic acid groups include carboxylic acid salt groups, carboxylic acid amide groups, carboxylic acid imide groups, carboxylic acid anhydride groups and carboxylic acid ester groups.

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Other examples of water-absorbent compositions can be found in US 4798861, WO93/17066, WO93/255735, WO 93/24684, WO93/12275, European Published Application Nos 0 401 044, 0 269 393, 0 326 382, 0 227 305, 0 101 253, 0 213 799, 0 232 121, 0 342 919, 0 233 014, 0 268 498 and 0 397 410, British Patent Application Nos 2 082 614, 2 022 505, 2 270 030, 2 269 602 and 2 126 591, US Patent Nos 4,418,163, 4,418,163, 3,989,586, 4,332,917, 4,338,417, 4,420,588 and 4,155,957 and French Patent

Application No. 2 525 121 which are all incorporated herein by reference.

The extrudable compositions of the invention are suitable for manufacture of substantially any article made using currently extrudable polymers, including films and bottles and any other articles. The compositions are suited to manufacture of filaments and fibre, for use in spunbond, non-woven and melt-blown applications. The composition is suitable also for manufacture of such articles as agri-chem sachets, mulch films, plant pots, domestic bags, diapers, drinking straws, fem care products, hangers, incontinence pads, sachets, six pack rings, disposable clothing, expanded foams, gloves, film canisters, golf tees, shot gun cartridges, bed pans, bottles, bowls, cotton buds, hospital curtains, "one-use" sterile products and packaging materials.

Compositions of PVA have good gas and fossil fuel barrier properties and are particularly suitable for making balloons and disposable packaging for oil and the like.

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Good results have been obtained with embodiments having the following make up, by weight:-

- (a) 40 to 80% PVA;
- (b) 5 to 50% filler;
- 20 © 5 to 15% plasticiser, preferably glycerol;
  - (d) 0.5 to 2.5% lubricant, preferably a fatty acid amide as an internal lubricant, most preferably octodecanamide.

Particularly good results have been obtained in embodiments of the invention having the following make-up, by weight:-

- (a) 40 to 70% PVA;
- (b) 20 to 50% filler, preferably a micronised inorganic material such as talc, preferably coated with stearate;
- © 8 to 15% plasticizer, preferably glycerol;
- 30 (d) 0.5 to 1.5% internal lubricant, preferably a fatty acid amide, most preferably octodecanamide; and

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(e) 0.0001 to 0.1% external lubricant, preferably stearate.

The composition may contain residual moisture, sufficient to enable the composition to be bound as a cold-pressed tablet.

5 Further advantageous results have been obtained in compositions of the invention comprising, by weight:-

50 to 60% PVA;

30 to 40% stearate-coated calcium carbonate;

8 to 15% glycerol;

0.5 to 1.5% octadecanamide; and

0.0001 to 0.1% zinc stearate.

In a third aspect, the invention provides a method of making an extrudable PVA-containing composition comprising blending PVA with a lubricant, said lubricant including a fatty acid amide. The method preferably comprises blending PVA with a fatty acid amide in the presence of an amount of moisture sufficient to bind the composition into tablets or pellets upon cold pressing.

The amount of lubricant blended with the PVA is typically restricted by the fact that, above a certain percent, excess lubricant separates out from the blend, and generally lubricant is blended at up to 5% by weight of the PVA present. The method thus conveniently enables tablets or pellets of the composition to be prepared as the feedstock for an extrusion. Blending of the components of the composition is achieved using conventional apparatus, one such example being a high speed blender.

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The method may optionally comprise adding small amounts of moisture to the components to be blended, such as by dripping water into the blend. Preferably, PVA and lubricant are fed into a high speed mixer gravimetrically.

30 In a fourth aspect, the invention provides a method of making a PVA-containing composition comprising blending PVA and a filler, wherein the filler comprises a

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superabsorbent material. It is preferred that blending is carried out in the presence of sufficient moisture to bind the composition into tablets or pellets upon cold pressing, the moisture optionally being present in the starting materials of the blend or being introduced into the blend, such as by drip-feeding.

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A fifth aspect of the invention provides a method of extruding a PVA-containing composition so as to form an extruded product which contains PVA, comprising:-

blending PVA with at least one component selected from a lubricant and a filler; and

cold pressing the resultant blend into pellets or tablets;

wherein the pellets or tablets comprise the polymer feedstock for extrusion of an extruded product containing PVA.

The PVA is preferably blended with one or more lubricant or filler components in the presence of sufficient moisture to bind the blend upon cold pressing. An advantage of the method is that the PVA-containing feedstock for the extrusion step is obtainable substantially without melting of the PVA. This improves the stability of the PVA during subsequent extrusion. Hitherto, as has been recognised in the present invention, attempts to extrude a PVA-containing feedstock largely failed as the PVA already had a heat history, ie had been wholly or partially melted in processing stages prior to the extrusion stage. The present invention avoids the disadvantage of introducing such a heat history into the PVA-containing composition.

A sixth aspect of the invention provides a method of extruding a PVA-containing composition comprising blending PVA and at least one optional component selected from a lubricant and a filler to form a feedstock for an extrusion process, the feedstock being in the form suitably of powder, pellets or tablets, and extruding the feedstock into a product, wherein the feedstock is prepared substantially without melting of the PVA.

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A seventh aspect of the invention provides a method of extruding a polymer composition containing a heat sensitive polymer, comprising blending the heat sensitive polymer with one or more optional components selected from a filler and a lubricant to form a feedstock for an extrusion process, the feedstock being suitably in the form of powder or pellets or tablets, and extruding the feedstock into an extruded product, wherein the feedstock is prepared substantially without melting of the heat sensitive polymer.

Optional and preferred features of the first and second aspects of the invention are, similarly, optional and preferred features of the third to seventh aspects of the invention.

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The invention is now described in the following non-limiting examples.

### Example 1

15 A blend of, by weight, approximately 60% fully hydrolysed PVA, 30% calcium carbonate, 10% glycerol, 0.01% zinc stearate and 1% octadecanamide was prepared in a high speed blender. It was found to have a white/cream colour with the following properties:-

density  $1.65 \text{ g/cm}^3$ 

melt density 1.46 g/cm<sup>3</sup> at 200°C (under ISO 1183)

MFR 357 (10 mins/200°C/21.6 kilograms, under ISO

1133)

melt temperature 200°C

25 processing temperature 190 - 200°C

residence time up to 15 minutes

drying time 4 hours at 80°C

### Example 2

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A blend was prepared in a similar way to example 1 of, by weight, approximately 60%

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partially hydrolysed PVA, 30% calcium carbonate, 10% glycerol, 0.01% zinc stearate and 1% octadecanamide. This was found to have the following properties:-

density 1.65 g/cm<sup>3</sup>

melt density 1.38-1.40 g/cm<sup>3</sup> at 190°C (under ISO 1183)

MFR 22 (10 mins/190°C/5 kilograms, under ISO 1133)

apparent melt viscosity 236/49 (Pa.s 1,000s/10,000s)

melt temperature 200°C

processing temperature 190-200°C

residence time up to 15 minutes

drying time 4 hours at 80°C

### Example 3

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- The PVA-containing compositions of examples 1 and 2 were examined for their extrudability in injection-moulding machines made by Brabender, Killion, Windsor, Hesas, Battenfield, Fischer, Demag and Arburg. Extrusion processing was carried out using a single full flight screw with constant pitch. The barrel temperature had a profile of 180-200°C (melt 190-210°C) and the screw speed varied typically between 20 and 120 rpm. Shut down of the apparatus was carried out by maintaining the temperature for up to 15 minutes with screw rotation stopped, over a period of 3 hours reducing the temperature to 100°C with screw rotation stopped and subsequently completing the shut down by turning the machine off.
- Whether or not a given composition is extrudable (by which is meant on commonplace extrusion apparatus) will normally be clear to one skilled in the art. In the event that a test is required, however, it is suggested that extrudability is determined by attempting extrusion on one or more of the above-named manufacturers machines using a single full flight screw with constant pitch with speeds and temperatures in the above range. If the composition extrudes reliably on at least two of the above machines with routine adjustment of parameters, it is to be deemed extrudable; if consistent problems are

obtained and extrusion is only possible under highly specific conditions or on specialised equipment, it is to be deemed not to be extrudable.

The compositions of both examples 1 and 2 were satisfactorily extruded into a range of films and tubes blow-moulded containers of various sizes and colours and injected moulded products.

### Examples 4-9

10 PVA-containing compositions were prepared as blends of the following components in the amounts shown:-

EXAMPLE							
	4	5	6	7	8	9	
Components							
PVA - fully hydrolysed	-	-	-	50	60	55	
PVA - partially	50	60	55	-	-	-	
hydrolysed							
Calcium carbonate	40	30	30	40	30	30	
(coated)							
Glycerol	9	9	14	9	9	14	
Octadecanamide	1	1	1	1	11	1	
Zinc Stearate	0.01	0.01	0.01	0.01	0.01	0.01	

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### Example 10

30 A further filled PVA-containing compositions was prepared as a blend of, by weight,

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approximately 60% partially hydrolysed PVA, 30% cross-linked sodium polyacrylate (a superabsorbent), 9% glycerol, 1% octadecanamide and 0.01% zinc stearate.

As far as extrusion apparatus is concerned, it is advantageous to use chrome plated screws and for the melt flow path to use chrome plated surfaces and gradual tapers to streamline the melt.

The invention thus provides PVA-containing compositions for use in the manufacture of bio-degradable articles. The invention extends to combinations of all features individually disclosed. In particular, optional or preferred features of any of the apparatus, product or method aspects may be combined with features of other aspects.



### **CLAIMS**

1. A PVA-containing composition, comprising a blend of PVA and an amount of lubricant effective to render the blend extrudable.

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- 2. A composition according to Claim 1 wherein the lubricant comprises a fatty acid amide.
- 3. A composition according to Claim 2 wherein the fatty acid amide is a straight or branched  $C_{12}$ - $C_{24}$  fatty acid amide.
  - 4. A composition according to Claim 2 or 3 wherein the composition further comprises a plasticiser.
- 15 5. A composition according to Claim 4 wherein the plasticiser is selected from ethylene glycol, glycerine, glycerol, triethylene glycol, low molecular weight polyethylene glycols and C<sub>2</sub>-C<sub>8</sub> amides.
- 6. A composition according to any of Claims 1 to 5 comprising up to 5% lubricant, 20 by weight.
  - 7. A composition according to any of Claims 1 to 6 comprising up to 20% plasticiser, by weight.
- 25 8. A composition according to any of Claims 1 to 7, in the form of a tablet or pellet obtainable by cold-pressing
  - 9. A filled PVA-containing composition, comprising a blend of PVA plus filler.
- 30 10. A composition according to Claim 9 wherein the filler comprises inert, inorganic material.



- 11. A composition according to Claim 9 wherein the filler comprises a superabsorbent material.
- 12. A composition according to Claim 9 wherein the filler comprises both an inert,5 inorganic material and superabsorbent material.
  - 13. A composition according to Claim 9, 10 or 12 wherein the inorganic filler comprises calcium carbonate.
- 10 14. A composition according to any of Claims 9-13 further comprising a lubricant.
  - 15. A composition according to Claim 14 wherein the lubricant comprises a fatty acid amide.
- 15 16. A composition according to any of Claims 14 to 15 comprising, by weight, up to 50% filler, up to 5% lubricant and up to 20% of a plasticizer.
  - 17. A composition according to any of Claims 14 to 16 comprising, by weight, 5-50% filler, 0.5 to 2.5% lubricant, 5 to 15% plasticizer and 40-80% PVA.
  - 18. A composition according to any of Claim 1-17, further comprising an additional lubricant component to reduce adhesion of the composition to surfaces.
  - 19. A composition according to Claim 18 wherein the lubricant comprises a stearate.
  - 20. A composition according to any of Claims 9 to 19 in the form of a tablet or pellet obtainable by cold-pressing.
  - 21. A PVA-containing composition comprising, by weight:-
- 30 40 to 80% PVA;

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5 to 50% filler;



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5 to 15% plasticizer; and

22. A composition according to Claim 21, comprising, by weight:-

40 to 70% PVA;

5 20 to 50% filler;

8 to 15% plasticizer;

0.5 to 2.5% lubricant, internal lubricant; and

0.0001 to 0.1% external lubricant.

10 23. A composition according to Claim 22 comprising, by weight:-

50 to 60% PVA;

30 to 40% stearate-coated calcium carbonate;

8 to 15% glycerol;

0.5 to 1.5% octadecanamide; and

0.0001 to 0.1% zinc stearate.

- 24. A method of making a PVA-containing composition comprising blending PVA with a lubricant, said lubricant including a fatty acid amide.
- 20 25. A method according to Claim 24 comprising blending PVA with a fatty acid amide in the presence of an amount of moisture sufficient to bind the composition into tablets or pellets upon cold pressing.
- 26. A method according to Claim 24 and 25 comprising blending, in a high speed blender, PVA with up to 5% by weight of lubricant.
  - 27. A method according to any of Claims 24 to 26 comprising adding moisture to the components to be blended, such as by dripping water into the blend.
- 30 28. A method according to any of Claims 24 to 27 wherein PVA and lubricant are fed into a high speed mixer gravimetrically.

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29. A method of making a PVA-containing composition comprising blending PVA and a filler, wherein the filler comprises a superabsorbent material.

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- 30. A method according to Claim 29 comprising blending PVA and a superabsorbent
   5 material in the presence of sufficient moisture to bind the composition into tablets or pellets upon cold pressing.
  - 31. A method of extruding a PVA-containing composition so as to form an extruded product which contains PVA, comprising:-

blending PVA with at least one optional component selected from a lubricant and a filler;

cold pressing the resultant blend into pellets or tablets;

wherein the pellets or tablets comprise the polymer feedstock for extrusion of an extruded product containing PVA.

- 32. A method according to Claim 31 comprising blending PVA with one or more
   20 optional components in the presence of sufficient moisture to bind the blend upon cold pressing.
  - 33. A method of extruding a PVA-containing composition comprising blending PVA and at least one optional component selected from a lubricant and a filler to form a feedstock for an extrusion process, the feedstock being in the form suitably of powder, pellets or tablets, and extruding the feedstock into a product, wherein the feedstock is prepared substantially without melting of the PVA.
- 34. A method of extruding a polymer composition containing a heat sensitive polymer,30 comprising blending the heat sensitive polymer with one or more optional components selected from a filler and a lubricant to form a feedstock for an extrusion process, the



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feedstock being suitably in the form of powder or pellets or tablets, and extruding the feedstock into an extruded product, wherein the feedstock is prepared substantially without melting of the heat sensitive polymer.

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 CO8K13/02 CO8L29/04

//(C08K13/02,5:20,3:26)

According to International Patent Classification (IPC) or to both national classification and IPC

### B. FIELDS SEARCHED

 $\begin{array}{lll} \mbox{Minimum documentation searched (classification system followed by classification symbols)} \\ IPC 7 & C08K & C08L \\ \end{array}$ 

Documentation searched other than manimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT						
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X Further documents are listed in the continuation of box C.	χ Patent family members are listed in annex.
<ul> <li>Special categories of cited documents:</li> <li>"A" document defining the general state of the lart which is not considered to be of particular relevance</li> <li>"E" earlier document but published on or after the international filling date</li> <li>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</li> <li>"O" document referring to an oral disclosure, use, exhibition or other means</li> <li>"P" document published prior to the international filling date but later than the priority date claimed</li> </ul>	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention  "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone  "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents. Such combination being obvious to a person skilled in the art.  "8" document member of the same patent family
Date of the actual completion of the international search  14 December 1999	Date of mailing of the international search report  22/12/1999
Name and mailing address of the ISA  European Patent Office. P.B. 5818 Patentlaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040. Tx. 31 651 epo nl.  Fax: (+31-70) 340-3016	Authorized officer  Engel, S

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Intc :onal Application No PCT/GB 99/02822

C.(Continu	ation) DOCUMENTS CONSIDERED TO BE RELEVANT	
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A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C08K13/02 C08L29/04

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According to International Patent Classification (IPC) or to both national classification and IPC

### B. FIELDS SEARCHED

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'Special categories of cited documents:  "A" document defining the general state of the lart which is not considered to be of particular relevance  "E" earlier document but published on or after the international filing date  "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)  "O" document referring to an oral disclosure, use. exhibition or other means  "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention.  "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone.  "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.  "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report  22/12/1999
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European Patent Office. P.B. 5818 Patentiaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040. Tx. 31 651 epo nl. Fax: (+31-70) 340-3016	Engel, S

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